

**WHAT IS CLAIMED IS:**

1. An O-substituted hydroxylamine free base having the following general formula:



- 5            wherein X is hydrogen or an alkyl; and R<sup>1</sup> is an unsubstituted or substituted phenyl, thienyl, furanyl, pyrrolyl or  $-\text{CR}^2=\text{CR}^3\text{R}^4$ ; wherein R<sup>2</sup>, R<sup>3</sup> and R<sup>4</sup> are hydrogen, halogen or alkyl; and

- 10           wherein said O-substituted hydroxylamine exhibits at least one property selected from the group consisting of:

- essentially free of hydroxylamine;  
essentially free of any solvent;  
a water content of between about 0% to 90% by weight; and  
15           a high strength (as measured by mole of said O-substituted hydroxylamine per gram of sample) of between about 0.5 to 3.3-fold as much as a 40% O-substituted hydroxylamine salt solution, by weight.

- 20           2. The O-substituted hydroxylamine according to claim 1, wherein said O-substituted hydroxylamine is at least one isomer selected from the group consisting of *cis*, *trans* and a mixture thereof.

- 25           3. The O-substituted hydroxylamine according to claim 1, wherein said O-substituted hydroxylamine has at least one additional property selected from the group consisting of:

a purity of between about 98% to 100%, based on gas chromatographic area; and

a purity drop of less than about 1.2% after 78 days at 40°C when said O-substituted hydroxylamine has a concentration of about 85% in water, based on gas chromatographic area.

- 5           4. The O-substituted hydroxylamine according to claim 1, wherein said O-substituted hydroxylamine is O-(3-chloro-2-propenyl)hydroxylamine.
- 10           5. The O-substituted hydroxylamine according to claim 1, wherein said O-substituted hydroxylamine is formed by the following steps comprising:
- 15           a. optionally admixing an organic solvent with a solution of O-substituted hydroxylamine salt to form at least an aqueous phase and extracting impurities from said aqueous phase;
- 20           b. optionally stripping residual organic solvents from said aqueous phase to form an aqueous-enriched phase;
- c. adjusting the pH of said aqueous-enriched phase to a pH of at least about 3.5, thereby forming an organic-enriched stream and an aqueous phase stream; and
- 25           d. separating said O-substituted hydroxylamine from said organic-enriched stream and/or said aqueous phase stream.
6. The O-substituted hydroxylamine according to claim 5, wherein said organic solvent used in extraction step (a) is selected from the group consisting of: aliphatic compounds, aromatic compounds, and alicyclic compounds.
- 30           7. The O-substituted hydroxylamine according to claim 5, wherein the extraction step (a) is conducted at a pH of about 7 or less.

8. The O-substituted hydroxylamine according to claim 7, wherein said pH is less than about 4.
- 5 9. The O-substituted hydroxylamine according to claim 5, wherein the extracting step (a) is conducted at a temperature between about  $-10$  to  $97^{\circ}\text{C}$ .
- 10 10. The O-substituted hydroxylamine according to claim 5, wherein said O-substituted hydroxylamine salt is O-substituted hydroxylamine hydrochloride.
- 15 11. The O-substituted hydroxylamine according to claim 10, wherein said O-substituted hydroxylamine hydrochloride is O-(3-chloro-2-propenyl)hydroxylamine hydrochloride.
12. The O-substituted hydroxylamine according to claim 5, wherein the extracting step (a) is conducted at a pressure between about 0 to 15 atmospheric pressure (atm).
- 20 13. The O-substituted hydroxylamine according to claim 5, wherein said separating step (d) comprises at least one step selected from the group consisting of:
- 25 (i) separating said organic-enriched stream from said aqueous phase stream, wherein said organic-enriched stream comprises said O-substituted hydroxylamine;
- (ii) distilling said aqueous stream, wherein said O-substituted hydroxylamine is taken overhead;
- 30 (iii) optionally, distilling said organic-enriched stream and said aqueous phase stream from the pH

adjustment step (c), wherein said O-substituted hydroxylamine is taken overhead; and  
(iv) optionally, fractionally distilling said organic-enriched stream, wherein a substantially solvent-free O-substituted hydroxylamine is taken overhead.

14. The O-substituted hydroxylamine according to claim 13, wherein said phase separation step (i) is conducted at a pH of between about 3.5 to 14.
15. The O-substituted hydroxylamine according to claim 14, wherein the pH during said phase separation step (i) is between about 4.5 to 13.
16. The O-substituted hydroxylamine according to claim 13, wherein the phase separation step (i) is conducted at a temperature of between about  $-10^{\circ}\text{C}$  to  $97^{\circ}\text{C}$ .
17. The O-substituted hydroxylamine according to claim 13, wherein the phase separation step (i) is conducted at a pressure between about 0 to 15 atm.
18. The O-substituted hydroxylamine according to claim 13, wherein said distilling step (ii) is conducted via distillation.
19. The O-substituted hydroxylamine according to claim 18, wherein said distillation comprises at least a column, a condenser, a reflux ratio controller, and a receiver.
20. The O-substituted hydroxylamine according to claim 19, wherein said column comprises about 0 to 35 theoretical plates.

21. The O-substituted hydroxylamine according to claim 20, wherein column comprises about 5 to 15 theoretical plates.
- 5 22. The O-substituted hydroxylamine according to claim 13, wherein said distilling step (ii) is conducted at a pressure between about 5 to 800 torr.
23. The O-substituted hydroxylamine according to claim 13, further comprising the addition of water during said distilling step (ii).
- 10 24. The O-substituted hydroxylamine according to claim 13, wherein said distilling step (iii) is conducted at a pressure between about 5 to 800 torr.
- 15 25. The O-substituted hydroxylamine according to claim 13, wherein said distilling step (iv) is conducted at a pressure between about 0 to 100 torr.
- 20 26. The O-substituted hydroxylamine according to claim 5, wherein said aqueous-enriched phase is adjusted to a pH between about 3.5 to 14.
27. The O-substituted hydroxylamine according to claim 26, wherein said pH of said aqueous-enriched phase is adjusted to between about 4.5 to 13.
- 25 28. The O-substituted hydroxylamine according to claim 27, wherein said pH of said aqueous-enriched phase is adjusted to between about 6.5 to 13.
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29. The O-substituted hydroxylamine according to claim 5, wherein said pH adjusting step (c) is conducted at a temperature between about –10°C to 97° C.
- 5        30. A method for forming O-substituted hydroxylamine comprising:
- a. optionally admixing an organic solvent with an O-substituted hydroxylamine salt to form at least an aqueous phase and extracting impurities from said aqueous phase;
  - 10        b. optionally stripping residual organics from said aqueous phase to form an aqueous-enriched phase;
  - c. adjusting the pH of said aqueous-enriched phase to a pH of at least about 3.5, thereby forming an organic-enriched stream and an aqueous phase stream; and
  - 15        d. separating said O-substituted hydroxylamine from said organic-enriched stream and/or said aqueous phase stream.
- 20        31. The method according to claim 30, wherein said organic solvent used in extraction step (a) is at least one selected from the group consisting of: aliphatic compounds, aromatic compounds, and alicyclic compounds.
- 25        32. The method according to claim 30, wherein the extraction step (a) is conducted at a pH of about 7 or less.
33. The method according to claim 28, wherein said pH is less than about 4.
- 30        34. The method according to claim 30, wherein the extracting step (a) is conducted at a temperature between about –10 to 97°C.

35. The method according to claim 30, wherein said O-substituted hydroxylamine salt is O-substituted hydroxylamine hydrochloride.

5 36. The method according to claim 35, wherein said O-substituted hydroxylamine hydrochloride is O-(3-chloro-2-propenyl)hydroxylamine hydrochloride.

37. The method according to claim 30, wherein the extracting step (a) is conducted at a pressure between about 0 to 15 atm.

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38. The method according to claim 30, wherein said separating step (d) is at least one step selected from the group consisting of:

- 15 (i) separating said organic-enriched stream from said aqueous phase stream, wherein said organic-enriched stream comprises said O-substituted hydroxylamine;
- (ii) distilling said aqueous stream, wherein said O-substituted hydroxylamine is taken overhead;
- 20 (iii) distilling said organic-enriched stream and said aqueous phase stream from the pH adjustment step (c), wherein said O-substituted hydroxylamine is taken overhead; and
- (iv) fractionally distilling said organic-enriched stream, wherein a substantially solvent-free O-substituted hydroxylamine is taken overhead.
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39. The method according to claim 38, further comprising the step of combining said organic-enriched stream from step (i) with the distillation overhead from either step (ii) or step (iii).

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40. The method according to claim 38, wherein said phase separation step (i) is conducted at a pH of about at least 3.5.
- 5 41. The method according to claim 40, wherein the pH during said phase separation step (i) is between about 4.5 to 13.
42. The method according to claim 38, wherein the phase separation step (i) is conducted at a temperature between about  $-10$  to  $97^{\circ}\text{C}$ .
- 10 43. The method according to claim 38, wherein the phase separation step (i) is conducted at a pressure between about 0 to 15 atmospheric pressure.
- 15 44. The method according to claim 38, wherein said distilling step (ii) is conducted via distillation.
45. The method according to claim 44, wherein said distillation comprises at least a column, a condenser, a reflux ratio controller, and a receiver.
- 20 46. The method according to claim 45, wherein said column comprises about 0 to 35 theoretical plates.
- 25 47. The method according to claim 46, wherein column comprises about 5 to 15 theoretical plates.
48. The method according to claim 38, wherein said distilling step (ii) is conducted at a pressure between about 5 to 800 torr.
- 30 49. The method according to claim 38, further comprising the addition of water during said distilling step (ii).



50. The method according to claim 30, wherein said pH of said aqueous-enriched phase is adjusted to between about 3.5 to 14.

5 51. The method according to claim 50, wherein said pH of said aqueous-enriched phase is adjusted to between about 4.5 to 13.

52. The method according to claim 30, wherein said pH adjusting step (c) is conducted at a temperature between about -10 to 97°C.

10 53. The method according to claim 30, wherein said O-substituted hydroxylamine has the following general formula:



15 wherein X is hydrogen or an alkyl; and R<sup>1</sup> is an unsubstituted or substituted phenyl, thienyl, furanyl, pyrrolyl or -CR<sup>2</sup>=CR<sup>3</sup>R<sup>4</sup>;  
wherein R<sup>2</sup>, R<sup>3</sup> and R<sup>4</sup> are hydrogen, halogen or alkyl.

54. The method according to claim 53, wherein said O-substituted hydroxylamine exhibits at least one property selected from the group consisting of:

20 essentially free of hydroxylamine;  
essentially free of any solvent;  
a water content of between about 0% to 90% by weight; and  
a high strength (as measured by mole of said O-substituted hydroxylamine per gram of sample) of between about 0.5 to 3.3-fold as much as a 40% O-substituted hydroxylamine salt solution, by  
25 weight.

55. The method according to claim 54, wherein said O-substituted hydroxylamine further comprising at least one additional property selected from the group consisting of:

a purity of between about 98% to 100%, based on gas chromatographic area; and

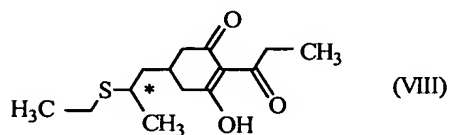
a purity drop of less than about 1.2% after 78 days at 40°C when said O-substituted hydroxylamine has a concentration of about 85% in water, based on gas chromatographic area.

56. The method according to claim 53, wherein said O-substituted hydroxylamine is at least one isomer selected from the group consisting of *cis*, *trans* and a mixture thereof.

57. The method according to claim 30, wherein said organic-enriched stream from said pH adjusting step (c) has a concentration of O-substituted hydroxylamine of between about 82-88%.

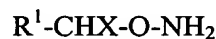
58. An herbicidal composition formed from the reaction product of:

a trione intermediate having the general formula:



; and

an O-substituted hydroxylamine having the following general formula:



wherein X is hydrogen or an alkyl; and R<sup>1</sup> is an unsubstituted or substituted phenyl, thienyl, furanyl, pyrrolyl or –

$CR^2=CR^3R^4$ ; wherein  $R^2$ ,  $R^3$  and  $R^4$  are hydrogen, halogen or alkyl.

59. The herbicidal composition according to claim 58, wherein said O-substituted hydroxylamine exhibits at least one property selected from the group consisting of:
- essentially free of hydroxylamine;  
essentially free of any solvent;  
a water content of between about 0% to 90% by weight; and  
a high strength (as measured by mole of said O-substituted hydroxylamine per gram of sample) of between about 0.5 to 3.3-fold as much as a 40% O-substituted hydroxylamine salt solution, by weight.
60. The herbicidal composition according to claim 59, wherein said O-substituted hydroxylamine further comprising at least one additional property selected from the group consisting of:
- a purity of between about 98% to 100%, based on gas chromatographic area; and  
a purity drop of less than about 1.2% after 78 days at 40°C when said O-substituted hydroxylamine has a concentration of about 85% in water, based on gas chromatographic area.
61. The herbicidal composition according to claim 58, wherein said O-substituted hydroxylamine is at least one isomer selected from the group consisting of *cis*, *trans* and a mixture thereof

62. The herbicidal composition according to claim 58, wherein said O-substituted hydroxylamine is formed by the following steps comprising:

- 5           a. optionally admixing an organic solvent with an O-substituted hydroxylamine salt to form at least an aqueous phase and extracting impurities from said aqueous phase;
- b. optionally stripping residual organics from said aqueous phase to form an aqueous-enriched phase;
- 10          c. adjusting the pH of said aqueous-enriched phase to a pH of at least about 3.5, thereby forming an organic-enriched stream and an aqueous phase stream; and
- d. separating said O-substituted hydroxylamine from said organic-enriched stream and/or said aqueous phase stream.

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63. The herbicidal composition according to claim 62, wherein said organic solvent is at least one selected from the group consisting of: aliphatic compounds, aromatic compounds, and alicyclic compounds.

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64. The herbicidal composition according to claim 62, wherein the extraction step (a) is conducted at a pH about 7 or less.

65. The herbicidal composition according to claim 64, wherein said pH is less than about 4.

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66. The herbicidal composition according to claim 62, wherein the extracting step (a) is conducted at a temperature between about -10 to 97°C.

67. The herbicidal composition according to claim 62, wherein said O-substituted hydroxylamine salt is O-substituted hydroxylamine hydrochloride.
- 5 68. The herbicidal composition according to claim 67, wherein said O-substituted hydroxylamine hydrochloride is O-(3-chloro-2-propenyl)hydroxylamine hydrochloride.
- 10 69. The herbicidal composition according to claim 62, wherein the extracting step (a) is conducted at a pressure between about 0 to 15 atm.
- 15 70. The herbicidal composition according to claim 62, wherein said separating step (d) is at least one step selected from the group consisting of:
- (i) separating said organic-enriched stream from said aqueous phase stream, wherein said organic-enriched stream comprises said O-substituted hydroxylamine;
  - 20 (ii) distilling said aqueous stream, wherein said O-substituted hydroxylamine is taken overhead;
  - (iii) distilling said organic-enriched stream and said aqueous phase stream from the pH adjustment step (c), wherein said O-substituted hydroxylamine is taken overhead; and
  - 25 (iv) fractionally distilling said organic-enriched stream, wherein a substantially solvent-free O-substituted hydroxylamine is taken overhead.
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71. The herbicidal composition according to claim 70, wherein said phase separation step (i) is conducted at a pH of about 3.5 to 14.
- 5 72. The herbicidal composition according to claim 70, wherein the phase separation step (i) is conducted at a temperature of between about -10 to 97°C.
- 10 73. The herbicidal composition according to claim 72, wherein the phase separation step (i) is conducted at a temperature of between about 0 to 60°C.
- 15 74. The herbicidal composition according to claim 70, wherein the phase separation step (i) is conducted at a pressure between about 0 to 15 atm.
- 20 75. The herbicidal composition according to claim 70, wherein said distilling step (ii) is conducted via distillation.
- 25 76. The herbicidal composition according to claim 75, wherein said distillation comprises at least a column, a condenser, a reflux ratio controller, and a receiver.
77. The herbicidal composition according to claim 76, wherein said column comprises about 0 to 35 theoretical plates.
78. The herbicidal composition according to claim 77, wherein column comprises about 5 to 15 theoretical plates.
- 30 79. The herbicidal composition according to claim 70, wherein said distilling step (ii) is conducted at a pressure between about 5 to 800 torr.

80. The herbicidal composition according to claim 70, further comprising the addition of water during said distilling step (ii).
- 5 81. The herbicidal composition according to claim 62, wherein said pH of said aqueous-enriched phase in step (c) is adjusted to above about 3.5.
82. The herbicidal composition according to claim 81, wherein said pH of said aqueous-enriched phase is adjusted to above about 4.5.
- 10 83. The herbicidal composition according to claim 62, wherein said pH adjusting step (c) is conducted at a temperature between about -10 to 97°C.
- 15 84. The herbicidal composition according to claim 83, wherein said pH adjusting step (c) is conducted at a temperature between about 0 to 60°C.
- 20 85. The herbicidal composition according to claim 62, wherein said organic-enriched stream from said pH adjusting step (c) has a concentration of O-substituted hydroxylamine of between about 82-88%.
- 25 86. The herbicidal composition according to claim 58, further comprising at least one additive selected from the group consisting of: crop oil concentrates, surfactants, fertilizers, emulsifiers, dispersing agents, foaming activators, foam suppressants, and correctives.
- 30 87. The herbicidal composition according to claim 58, further comprising an inert carrier.

88. The herbicidal composition according to claim 58, further comprising at least one other biologically active chemical.

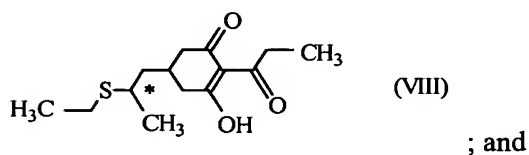
89. A method for controlling growth of vegetation comprising:

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applying to said vegetation a herbicidal composition formed from the reaction product of:

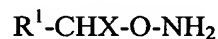
a trione intermediate having the general formula:

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an O-substituted hydroxylamine having the following general formula:

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wherein X is hydrogen or an alkyl; and  $R^1$  is an unsubstituted or substituted phenyl, thienyl, furanyl, pyrrolyl or  $-CR^2=CR^3R^4$ ; wherein  $R^2$ ,  $R^3$  and  $R^4$  are hydrogen, halogen or alkyl.

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90. The method according to claim 89, wherein said O-substituted hydroxylamine exhibits at least one property selected from the group consisting of:

essentially free of hydroxylamine;

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essentially free of any solvent;

a water content of between about 0% to 90% by weight; and



a high strength (as measured by mole of said O-substituted hydroxylamine per gram of sample) of between about 0.5 to 3.3-fold as much as a 40% O-substituted hydroxylamine salt solution, by weight.

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91. The method according to claim 90, wherein said O-substituted hydroxylamine further comprising at least one additional property selected from the group consisting of:

10 a purity of between about 98% to 100%, based on gas chromatographic area; and  
a purity drop of less than about 1.2% after 78 days at 40°C when said O-substituted hydroxylamine has a concentration of about 85% in water, based on gas chromatographic area.

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92. The method according to claim 89, wherein said O-substituted hydroxylamine is at least one isomer selected from the group consisting of *cis*, *trans* and a mixture thereof.

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93. The method according to claim 89, wherein said O-substituted hydroxylamine is formed by the following steps comprising:

- 25 a. optionally admixing an organic solvent with an O-substituted hydroxylamine salt to form at least an aqueous phase and extracting impurities from said aqueous phase;  
b. optionally stripping residual organic solvents from said aqueous phase to form an aqueous-enriched phase;  
c. adjusting the pH of said aqueous-enriched phase to a pH of at least about 3.5, thereby forming an organic-enriched stream and an aqueous phase stream; and  
30 d. separating said O-substituted hydroxylamine from said organic-enriched stream and/or said aqueous phase stream.

94. The method according to claim 93, wherein said separating step (d) is at least one step selected from the group consisting of:

- (i) separating said organic-enriched stream from said aqueous phase stream, wherein said organic-enriched stream comprises said O-substituted hydroxylamine;
- (ii) distilling said aqueous stream, wherein said O-substituted hydroxylamine is taken overhead;
- (iii) distilling said organic-enriched stream and said aqueous phase stream from the pH adjustment step (c), wherein said O-substituted hydroxylamine is taken overhead; and
- (iv) fractionally distilling said organic-enriched stream, wherein a substantially solvent-free O-substituted hydroxylamine is taken overhead.

95. The method according to claim 89, wherein said herbicidal composition further comprises at least one additive selected from the group consisting of: crop oil concentrates, surfactants, fertilizers, emulsifiers, dispersing agents, foaming activators, foam suppressants, and correctives.

96. The method according to claim 89, wherein said herbicidal composition further comprises an inert carrier.

97. The method according to claim 89, wherein said herbicidal composition further comprises at least one other biologically active chemical.

98. An O-substituted hydroxylamine free base having the following general formula:



wherein X is hydrogen or an alkyl; and  $R^1$  is an unsubstituted or substituted phenyl, thienyl, furanyl, pyrrolyl or  $-\text{CR}^2=\text{CR}^3\text{R}^4$ ; wherein  $R^2$ ,  $R^3$  and  $R^4$  are hydrogen, halogen or alkyl; and

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wherein said O-substituted hydroxylamine is essentially free of hydroxylamine.

99. An O-substituted hydroxylamine free base having the following general formula:

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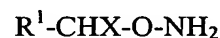
wherein X is hydrogen or an alkyl; and  $R^1$  is an unsubstituted or substituted phenyl, thienyl, furanyl, pyrrolyl or  $-\text{CR}^2=\text{CR}^3\text{R}^4$ ; wherein  $R^2$ ,  $R^3$  and  $R^4$  are hydrogen, halogen or alkyl; and

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wherein said O-substituted hydroxylamine is essentially free of any solvent.

100. An O-substituted hydroxylamine free base having the following general formula:

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wherein X is hydrogen or an alkyl; and  $R^1$  is an unsubstituted or substituted phenyl, thienyl, furanyl, pyrrolyl or  $-\text{CR}^2=\text{CR}^3\text{R}^4$ ; wherein  $R^2$ ,  $R^3$  and  $R^4$  are hydrogen, halogen or alkyl; and

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wherein said O-substituted hydroxylamine has a water content of between about 0% to 90% by weight.

101. An O-substituted hydroxylamine free base having the following general formula:



5 wherein X is hydrogen or an alkyl; and  $R^1$  is an unsubstituted or substituted phenyl, thienyl, furanyl, pyrrolyl or  $-\text{CR}^2=\text{CR}^3\text{R}^4$ ; wherein  $R^2$ ,  $R^3$  and  $R^4$  are hydrogen, halogen or alkyl; and

10 wherein said O-substituted hydroxylamine has a high strength (as measured by mole of said O-substituted hydroxylamine per gram of sample) of between about 0.5 to 3.3-fold as much as a 40% O-substituted hydroxylamine salt solution, by weight.

102. The O-substituted hydroxylamine according to claim 3, wherein said O-substituted hydroxylamine is O-(3-chloro-2-propenyl)hydroxylamine.
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103. The O-substituted hydroxylamine according to claim 5, wherein said aqueous-enriched phase in step (c) is an O-substituted hydroxylamine salt.
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104. The O-substituted hydroxylamine according to claim 103, wherein said O-substituted hydroxylamine salt is O-substituted hydroxylamine hydrochloride.

- 25 105. The O-substituted hydroxylamine according to claim 104, wherein said O-substituted hydroxylamine hydrochloride is O-(3-chloro-2-propenyl)hydroxylamine hydrochloride.

- 30 106. The method according to claim 53, wherein said O-substituted hydroxylamine is O-(3-chloro-2-propenyl)hydroxylamine.

107. The method according to claim 55, wherein said O-substituted hydroxylamine is O-(3-chloro-2-propenyl)hydroxylamine.
- 5 108. The method according to claim 30, wherein said aqueous-enriched phase in step (c) is an O-substituted hydroxylamine salt.
109. The method according to claim 108, wherein said O-substituted hydroxylamine salt is O-substituted hydroxylamine hydrochloride.
- 10 110. The method according to claim 109, wherein said O-substituted hydroxylamine hydrochloride is O-(3-chloro-2-propenyl)hydroxylamine hydrochloride.
- 15 111. The herbicidal composition according to claim 59, wherein said O-substituted hydroxylamine is O-(3-chloro-2-propenyl)hydroxylamine.
- 20 112. The herbicidal composition according to claim 60, wherein said O-substituted hydroxylamine is O-(3-chloro-2-propenyl)hydroxylamine.
- 25 113. The herbicidal composition according to claim 62, wherein said aqueous-enriched phase in step (c) is an O-substituted hydroxylamine salt.
114. The herbicidal composition according to claim 113, wherein said O-substituted hydroxylamine salt is O-substituted hydroxylamine hydrochloride.

115. The herbicidal composition according to claim 114, wherein said O-substituted hydroxylamine hydrochloride is O-(3-chloro-2-propenyl)hydroxylamine hydrochloride.

5 116. The method according to claim 89, wherein said O-substituted hydroxylamine is O-(3-chloro-2-propenyl)hydroxylamine.

117. The method according to claim 90, wherein said O-substituted hydroxylamine is O-(3-chloro-2-propenyl)hydroxylamine.

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118. The method according to claim 91, wherein said O-substituted hydroxylamine is O-(3-chloro-2-propenyl)hydroxylamine.

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119. The method according to claim 93, wherein said aqueous-enriched phase in step (c) is an O-substituted hydroxylamine salt.

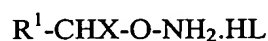
120. The method according to claim 119, wherein said O-substituted hydroxylamine salt is O-substituted hydroxylamine hydrochloride.

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121. The method according to claim 120, wherein said O-substituted hydroxylamine hydrochloride is O-(3-chloro-2-propenyl)hydroxylamine hydrochloride.

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122. An O-substituted hydroxylamine salt having the following general formula:



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wherein L is a halogen or hydrogensulfate; X is hydrogen or alkyl; R<sup>1</sup> is unsubstituted or substituted phenyl, thienyl, furanyl, pyrrolyl or –CR<sup>2</sup>=CR<sup>3</sup>R<sup>4</sup>; wherein R<sup>2</sup>, R<sup>3</sup>, and R<sup>4</sup> are each hydrogen, halogen or alkyl; and

wherein said O-substituted hydroxylamine salt is essentially free of hydroxylamine.

5           123.   The O-substituted hydroxylamine salt according to claim 122,  
              wherein said O-substituted hydroxylamine salt is at least one isomer  
              selected from the group consisting of *cis*, *trans* and a mixture thereof.

              124.   The O-substituted hydroxylamine salt according to claim 122,  
10                wherein said O-substituted hydroxylamine salt is O-(3-chloro-2-  
                  propenyl)hydroxylamine hydrochloride.

              125.   The O-substituted hydroxylamine salt according to claim 122,  
              wherein said O-substituted hydroxylamine salt is formed by the  
              following steps comprising:

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a.   optionally admixing an organic solvent with an hydroxylamine-  
containing O-substituted hydroxylamine salt to form at least an  
aqueous phase and extracting impurities from said aqueous phase;

20           b.   optionally stripping residual organic solvents from said aqueous  
              phase to form an aqueous-enriched phase;

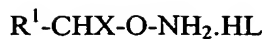
c.   adjusting the pH of said aqueous-enriched phase to a pH of at  
least about 3.5, thereby forming an organic-enriched stream and an  
aqueous phase stream;

25           d.   separating said O-substituted hydroxylamine from said organic-  
enriched stream and/or said aqueous phase stream; and

e.   reacidifying said O-substituted hydroxylamine, thereby forming  
said O-substituted hydroxylamine salt which is essentially free of  
hydroxylamine.

126. The O-substituted hydroxylamine salt according to claim 125,  
wherein step (e) is conducted at a temperature of about 75°C or less.
127. The O-substituted hydroxylamine salt according to claim 126,  
5 wherein step (e) is conducted at a temperature of about 65°C or less.
128. A method for forming O-substituted hydroxylamine salt  
comprising:
- 10 a. optionally admixing an organic solvent with an hydroxylamine-  
containing O-substituted hydroxylamine salt to form at least an  
aqueous phase and extracting impurities from said aqueous phase;  
b. optionally stripping residual organic solvents from said aqueous  
phase to form an aqueous-enriched phase;
- 15 c. adjusting the pH of said aqueous-enriched phase to a pH of at  
least about 3.5, thereby forming an organic-enriched stream and an  
aqueous phase stream;
- d. separating said O-substituted hydroxylamine from said organic-  
enriched stream and/or said aqueous phase stream; and
- 20 e. reacidifying said O-substituted hydroxylamine, thereby forming  
said O-substituted hydroxylamine salt which is essentially free of  
hydroxylamine.
129. The method according to claim 128, wherein step (e) is conducted  
25 at a temperature of about 75°C or less.
130. The method according to claim 129, wherein step (e) is conducted  
at a temperature of about 65°C or less.
131. The method according to claim 128, wherein said O-substituted  
30 hydroxylamine salt has the following general formula:





wherein L is a halogen or hydrogensulfate; X is hydrogen or alkyl; R<sup>1</sup> is unsubstituted or substituted phenyl, thienyl, furanyl, pyrrolyl or – CR<sup>2</sup>=CR<sup>3</sup>R<sup>4</sup>; wherein R<sup>2</sup>, R<sup>3</sup>, and R<sup>4</sup> are each hydrogen, halogen or alkyl; and

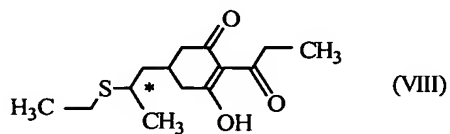
wherein said O-substituted hydroxylamine salt is essentially free of hydroxylamine.

132. The method according to claim 131, wherein said O-substituted hydroxylamine salt is at least one isomer selected from the group consisting of *cis*, *trans* and a mixture thereof.

133. The method according to claim 128, wherein said O-substituted hydroxylamine salt is O-(3-chloro-2-propenyl)hydroxylamine hydrochloride.

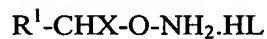
134. A herbicidal composition formed from the reaction product of:

a trione intermediate having the general formula:



; and

O-substituted hydroxylamine salt has the following general formula:



wherein L is a halogen or hydrogensulfate; X is hydrogen or alkyl; R<sup>1</sup> is unsubstituted or substituted phenyl, thienyl, furanyl, pyrrolyl or –

$CR^2=CR^3R^4$ ; wherein  $R^2$ ,  $R^3$ , and  $R^4$  are each hydrogen, halogen or alkyl; and

wherein said O-substituted hydroxylamine salt is essentially free of hydroxylamine.

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135. The herbicidal composition according to claim 134, wherein said O-substituted hydroxylamine salt is at least one isomer selected from the group consisting of: *cis*, *trans* and a mixture thereof

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136. The herbicidal composition according to claim 134, wherein said O-substituted hydroxylamine salt is formed by the following steps comprising:

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a. optionally admixing an organic solvent with an O-substituted hydroxylamine salt to form at least an aqueous phase and extracting impurities from said aqueous phase;

b. optionally stripping residual organics from said aqueous phase to form an aqueous-enriched phase;

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c. adjusting the pH of said aqueous-enriched phase to a pH of at least about 3.5, thereby forming an organic-enriched stream and an aqueous phase stream;

d. separating said O-substituted hydroxylamine from said organic-enriched stream and/or said aqueous phase stream; and

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e. reacidifying said O-substituted hydroxylamine, thereby forming said O-substituted hydroxylamine salt which is essentially free of hydroxylamine.

137. The herbicidal composition according to claim 136, wherein said separating step (d) is at least one step selected from the group consisting of:

- 5 (i) separating said organic-enriched stream from said aqueous phase stream, wherein said organic-enriched stream comprises said O-substituted hydroxylamine;
- (ii) distilling said aqueous stream, wherein said O-substituted hydroxylamine is taken overhead;
- 10 (iii) distilling said organic-enriched stream and said aqueous phase stream from the pH adjustment step (c), wherein said O-substituted hydroxylamine is taken overhead; and
- 15 (iv) fractionally distilling said organic-enriched stream, wherein a substantially solvent-free O-substituted hydroxylamine is taken overhead.

20 138. The herbicidal composition according to claim 134, further comprising at least one additive selected from the group consisting of: crop oil concentrates, surfactants, fertilizers, emulsifiers, dispersing agents, foaming activators, foam suppressants, and correctives.

25 139. The herbicidal composition according to claim 134, further comprising an inert carrier.

140. The herbicidal composition according to claim 134, further comprising at least one other biologically active chemical.

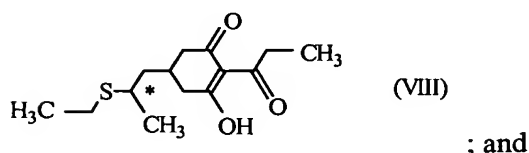
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141. A method for controlling growth of vegetation comprising:

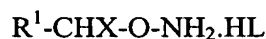
applying to said vegetation a herbicidal composition formed from the reaction product of:

a trione intermediate having the general formula:

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O-substituted hydroxylamine salt has the following general formula:



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wherein L is a halogen or hydrogensulfate; X is hydrogen or alkyl;  $R^1$  is unsubstituted or substituted phenyl, thienyl, furanyl, pyrrolyl or  $\text{-CR}^2=\text{CR}^3\text{R}^4$ ; wherein  $R^2$ ,  $R^3$ , and  $R^4$  are each hydrogen, halogen or alkyl; and

15

wherein said O-substituted hydroxylamine salt is essentially free of hydroxylamine.

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142. The method according to claim 141, wherein said O-substituted hydroxylamine salt is at least one isomer selected from the group consisting of *cis*, *trans* and a mixture thereof.

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143. The method according to claim 141, wherein said O-substituted hydroxylamine salt is formed by the following steps comprising:

a. optionally admixing an organic solvent with an O-substituted hydroxylamine salt to form at least an aqueous phase and extracting impurities from said aqueous phase;

- b. optionally stripping residual organic solvents from said aqueous phase to form an aqueous-enriched phase;
- c. adjusting the pH of said aqueous-enriched phase to a pH of at least about 3.5, thereby forming an organic-enriched stream and an aqueous phase stream;
- d. separating said O-substituted hydroxylamine from said organic-enriched stream and/or said aqueous phase stream; and
- e. reacidifying said O-substituted hydroxylamine, thereby forming said O-substituted hydroxylamine salt which is essentially free of hydroxylamine.
144. The method according to claim 143, wherein said separating step (d) is at least one step selected from the group consisting of:
- (i) separating said organic-enriched stream from said aqueous phase stream, wherein said organic-enriched stream comprises said O-substituted hydroxylamine;
- (ii) distilling said aqueous stream, wherein said O-substituted hydroxylamine is taken overhead;
- (iii) distilling said organic-enriched stream and said aqueous phase stream from the pH adjustment step (c), wherein said O-substituted hydroxylamine is taken overhead; and
- (iv) fractionally distilling said organic-enriched stream, wherein a substantially solvent-free O-substituted hydroxylamine is taken overhead.
145. The method according to claim 141, wherein said herbicidal composition further comprises at least one additive selected from the group consisting of: crop oil concentrates, surfactants, fertilizers, emulsifiers, dispersing agents, foaming activators, foam suppressants, and correctives.

146. The method according to claim 141, wherein said herbicidal composition further comprises an inert carrier.

5 147. The method according to claim 141, wherein said herbicidal composition further comprises at least one other biologically active chemical.

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